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Low Temperature Characteristics of Greases

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The strategic importance in military operations of an adequate supply of lubricants satisfactory for operation under extremes of temperature and humidity has prompted close study of many phases of the problem. Good lubricating qualities, mechanical and chemical stability at high and low temperatures without oil evaporation or separation, resistance to deterioration by moisture, and minimum change of effective consistency with temperature are the chief desiderata. While generally speaking any one grease might be called upon to function under such extremes of conditions, it is a matter of utmost importance to the military, and has long been a desire of the trade, that the full range of service should be covered by a minimum number of lubricants.

One specific phase of service requiring a special lubricant has been the exposed bearings of military aircraft. These may alternately be roasted under tropical suns and chilled to perhaps one hundred degrees below zero on nocturnal flights at high levels.

The study covered in this paper was undertaken as background for fabrication of lubricants suitable for such service, to establish the role of different soap bases and different oil viscosities in determining the behavior of greases in ball bearing service at low temperatures.

To evaluate the performance of the test lubricants at low temperatures, the torque test outlined in Army-Navy Specification AN-G-3 of May 30, 1942, was used. An eight ball 204 open bearing was packed with five grams of grease, closed with slip fit shields, and run in one hundred turns each way. The bearing was then chilled on a straight line curve to test temperature in two hours and soaked for one hour, rotating two turns every fifteen minutes. At the end of the soaking period the time necessary

for a complete revolution with 2000 gmcm applied torque was determined.

The apparatus used, shown in Figures 1, 2, and 3, consisted of a cold bath, a block for supporting test bearings, spindles, a fan, thermo-couples, and accessories for producing and measuring temperature and torque. The cold bath comprised a cylindrical one gallon silvered glass vacuum jug inside a protective wooden case. An oak block eight inches square and eight inches long was turned down along seven inches of its length to a cylinder which fitted loosely into the vacuum jug, that is, about six inches diameter. The eight inch square top overhanging the cylinder fitted snugly into the outer wooden case flush with its top and rested on an inside shelf so that the block was held in fixed position not touching the jug. This block was drilled for four vertical spindles, symmetrically placed as shown, a centrally placed fan shaft, a small inlet and outlet for refrigerant, and a tube for thermocouple leads.

Each test bearing was held in place by three steel pins projecting from the block. The outer race of each bearing was notched in one place sufficiently that the pressure of a pin at this point prevented rotation. These pins projected an inch from the block and the bearings were supported 1/2-inch from the block. The cylindrical spindles were of oak with metal caps at the cold end so threaded that the inner race of the test bearing could be held tight by a lock nut. The upper spindle guide bearing was held permanently in place by large screws and was packed with a very light grease. The top inch of the spindle was square in cross section to accommodate a four centimeter pulley with square hole to match. A small glass tube housed the eight thermocouple leads, while two 1/4-inch glass tubes served as inlet and outlet for refrigerant. The small fan shaft was housed in a glass tube shrunk to bearing clearances at either end. This was powered first by motor and pulley and then more conveniently by a small air turbine. A portable potentiometer was used to measure the thermocouple voltage, with the usual ice bath cold junction.

Refrigeration was achieved and controlled manually by admitting small increments of liquid nitrogen directly to the bottom of the cold bath where it immediately boiled to a gas absorbing heat from the air which was swirled rapidly about the bearings. Since the annular spaces about the block and the spindles were sealed at the top the dead gas space was an effective insulator; the thermal leakage was small in proportion to the thermal capacity; and close control was possible, while the low heat capacity permitted close control of the rate of cooling. The temperature range was limited only by the temperature of liquid nitrogen some 320° F. below zero. For flexibility, range, and freedom from moisture condensation the unit compared most favorably with carbon dioxide cooled units.

Another piece of apparatus which proved of interest in this study was one patterned after a unit described in the British Air Ministry's Specification DTD 201, "Controls Lubricating Oil." We constructed a modified multiple bearing unit and did some preliminary work which proved interesting. We discontinued this test, however, in favor of establishing a foundation with the standard test, to which we might later correlate results of the simpler unit.

Our modification consisted of a ½-inch steel ball on the end of a ½-inch shaft, seated into a brass socket bearing. The socket was held by a rubber sleeve in the flared end of a glass condenser tube, the upper end of



which was shrunk to a bearing fit about the 1/8-inch shaft. A pulley on the shaft above the tube served for applying torque and supporting the loading weights when used. We used four such units mounted vertically thru a lid which fitted the cold bath used for the ball bearing test. For coolant we used a liquid bath of acetone chilled with dry ice.

Our preliminary work indicated that the torque characteristics of greases in the range of useful consistencies was of the same order of magnitude as that of the base oil from which they were fabricated.

The lubricants tested in the ball bearing torque apparatus comprised greases of from 310 to 340 A.S.T.M. worked penetration made from six metallic bases and four base oils. Test samples were prepared as nearly free as possible from fibrous or granular structure and were of anhydrous type. No mixtures were tried. The fatty material used in compounding the greases was not identical in all cases, but was varied according to the requirements of the individual metal bases.

The oil designated as 100 viscosity was a conventionally refined naphthenic stock from a Louisiana crude and had a viscosity index of 25, a flash of 325° F., and a pour of -60° F. The 57 viscosity oil was of similar type, lower in viscosity and flash, with a pour point of -65° F. The 40 viscosity stock was a highly refined stock of 310° F. flash and -65° F. pour. The 34 viscosity oil was a gas oil from a local crude.

For the purpose of this testing the volatility characteristics of such light stocks were not considered. The object was to find the range of stocks suitable from the standpoint of low temperature performance without regard to other characteristics.

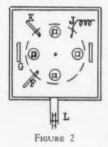
In TABLE I are shown the time of revolution of test bearings at the temperatures indicated. Very high and very low values are to be taken only as indicative of the order of magnitude involved.

The relation between time of revolution and oil viscosity for the various greases at 67° F. is shown graphically in Figure 4. Bearing in mind the inaccuracy of the test measurement for very stiff greases, the dispersion at the higher oil viscosities is not to be taken too literally. With the exception of the Barium grease the torque values are similar for the different bases to a degree within the limits of control of manufacture. Figure 5 represents an expansion of a limited area of Figure 4. Since a time of about thirty seconds is the maximum permissible in a satisfactory grease, the use of an oil of viscosity less than about 60 at 100° F. is indicated. For Barium greases an even lighter oil might be necessary. Many other factors might play a part in the choice. No tests were run on high soap content greases milled down to the desired consistency, nor on fibrous greases. Preliminary tests on Sodium base greases incorporating small amounts of organic esters seemed to indicate that such products were effective for reducing torque only as they reduced the effective oil viscosity. Figure 6 shows the logarithm of the time of revolution plotted against the oil viscosity at 100° F. The nearly linear nature of the curve indicates some logarithmic relationship between torque and viscosity. Figure 7 depicts the relationship between the time of revolution at -100° F. and oil viscosity at 100° F. of three types of grease. The relative unimportance of the metal base in determining torque characteristics is still apparent.

The four greases tested at -120° F. and -150° F. appear at first glance to be very different in torque characteristics; but, since they were all very satisfactory at -120° F. and there is currently little interest in greases functioning at -150° F., it may be assumed that the limiting oil viscosity for greases satisfactory at -120° F. will be somewhere in the neighborhood of 36 to 40 seconds at

In summary it may be stated the low temperature bearing torque characteristics of greases fabricated from different metal bases and low viscosity index oils appeared to be dependent chiefly on the viscosity of the oil used. Barium greases seemed to stiffen somewhat above the average of the other greases tested.

Since the use of very light oils is dictated by low temperature requirements, part of the problem lies with the refiner to produce stocks of the best possible volatility characteristics for the grease compounder to try his art on. Much work remains to be done in determining the effect of viscosity index, of phenomenal pour points, of blending agents, and of extreme pressure and/or oiliness additives which may be added to light oils.



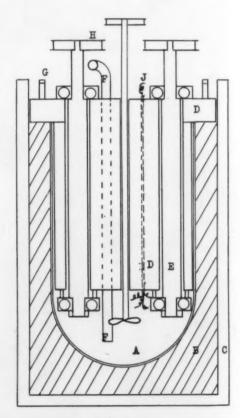


Figure 1
(Cuts continued on page 3)

TABLE I

Time in S	seconds for	revolution	of	test	bearing :	at	indicated	temperature
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Temp.	Oil Viscosity Saybolt @100°	Aluminum Base	Sodium Base	Lithium Base	Lead Base	Calcium Base	Barium Base
-67°F	100	950	507	535		653	1950
−67°F	57	17	17	19	16	22	55
-67°F	40	4	*****	<1	<1		
−67°F	34	<1	(1	(1	(1	(1	<1
-100°F	57		600	600	Frozen		
-100°F	40			85	92	*****	*****
-100°F	34	<1	<1	<1	1		1
-120°F	34	31/2	1/2	4			31/2
-150°F	34	502	182	420			1300

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November 2, 1942

PUBLICATION GIVES A.S.T.M. STANDARDS ON PETROLEUM PRODUCTS AND LUBRICANTS

Issued annually since 1927 and giving in their latest approved form all of the A.S. T.M. specifications, tests, and definitions pertaining to petroleum products and lubricants this publication sponsored by the A.S.T.M. Committee D-2 has come into very widespread use.

The 1942 (October) edition includes some 87 standards; in addition there are several proposed tests not yet adopted by the committee covering the following: Oil content of paraffin wax, color of lubricating oil by means of photoelectric colorimeter, potential gum in aviation gasoline, and oxidation characteristics of heavy-duty crankcase oils.

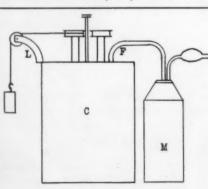


FIGURE 3

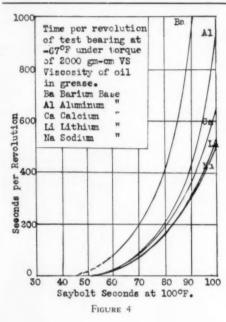
New standards given in the volume for the first time cover tests for neutralization number of petroleum products by color-indicator titration and by electrometric titration, test for rust-preventing characteristics of steamturbine oil in the presence of water, conversion of kinematic viscosity to saybolt furol viscosity, and a test for sludge formation in mineral transformer oil.

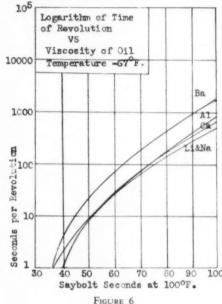
Included among the widely used standard tests for petroleum products are the following: Acid heat (gasoline), aniline point, burning quality (kerosine, long-time burning, and mineral seal oil), carbon residue, cloud and pour points, consistency (greases and petrolatum), ductility (bituminous materials), flash points by open cup, Pensky-Martens and Tag-closed tester, gum content (gasoline), and knock characteristics (motor and aviation fuels).

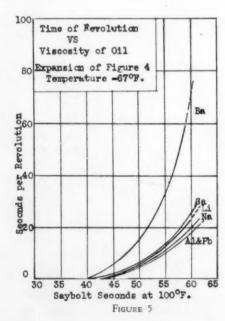
Also the following: Oxidation stability (gasoline), precipitation number, saponification number, sediment (fuel oil), tetraethyl lead (gasoline), thermal value (fuel oil), vapor pressure (motor, aviation, and natural gasoline), and water and sediment.

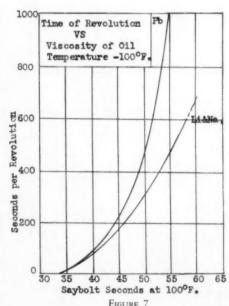
Specifications.—Specification requirements are given for cut-back asphalts (rapid-curing and medium-curing type), fuel oils, emulsified asphalts (four types), gasoline, petroleum spirits, stoddard solvent, and A.S.T.M. thermometers.

Copies of this 450-page publication can be obtained in heavy paper cover from A.S.T.M. Headquarters, 260 S. Broad St., Philadelphia, Pa., at \$2.25 per copy.









Flow Characteristics of Lubricating Greases

by

A. Beerbower (1), L. W. Sproule (2), J. B. Patberg (1), and J. C. Zimmer (1)

Presented before the Tenth Annual Convention of the National Lubricating Grease Institute

Petroleum lubricating greases, consisting of soap thickened mineral oils, are employed extensively for the lubrication of rubbing surfaces on all types of machinery and transport equipment. Their solid, or semi-solid, structure is advantageous from the standpoint of application to many types of bearings, and in addition it enables them to remain in mechanisms or lubricant reservoirs which are not oil or liquid tight, and to feed to the rubbing surfaces only when actual bearing movement occurs.

Grease thus serves to supply adequate lubrication to bearings with, at the most, only occasional attention to replenishment of lubricant supply. A simple grease fitting or cup, plus the space for a supply of greases in the bearing housing can, and frequently does, therefore, take the place of the comparatively expensive and cumbersome reservoir and distribution system required for oil bath lubrication. Common-place examples of such applications of greases are automotive chassis lubrication fittings requiring lubricant replenishment only at infrequent intervals; and the factory, life-time greased and sealed, anti-friction bearings in a household vacuum cleaner.

While the convenience in application and the reduced leakage and consumption inherent in the semi-solid consistency of greases is primarily responsible for their use as lubricants, it has been recognized for many years that the actual lubrication of rubbing surfaces must be accomplished by a fluid film. The proper type of greases will actually provide a more or less fluid lubricant film between rubbing surfaces, and thus offer a minimum of frictional drag. The resistance to flow of normal mineral oils is termed the viscosity, and this is one of the most important properties of an oil from the lubrication standpoint. The measurement of the viscosity of lubricating oils is relatively simple, since, while viscosity varies with the temperature, it is substantially independent of total pressure over an extremely wide

In the case of greases, however, the analogous property, which is usually termed the apparent viscosity or conversely, the mobility is dependent not only on temperature, but also varies with the pressure differential or the shearing stress, and the rate of shear. While either shearing stress versus shear rate, or apparent viscosity versus shear rate

graphs may be employed to show the change in viscosity, the latter is more commonly used. The rate of shear may be defined as the ratio of the velocity of flow in cm./sec. to the clearance between two parallel surfaces moving in opposite directions. In the case of a tube or capillary, this theoretically becomes four times the volume of flow in cm./sec. divided by πR^3 , where R is the radius of the capillary. Arveson (1) gives the rate of shear encountered in a 2" concentric journal and sleeve bearing, and points out the significance of rate of shear in dispensing greases as follows:

- (1) Research Division, Esso Laboratories, Standard Oil Development Company
- (2) Technical and Research Division, Imperial Oil, Ltd.

TABLE I
"Rate of Shear in 2-Inch Concentric Journal
Bearing Rotating at 1800 RPM"

Clear	ance	Rate of Shear	
Inch	(Cm.)	Seconds-1	
0.01	(0.025)	18,800	
0.001	(0.0025)	188,000	
0.0001	(0.00025)	1,880,000	

"In the simple case of a material flowing through a tube of ½" (0.32-cm.) bore at a rate of 3 ccm. per second (0.4 pound per minute), the rate of shear is approximately 1000 seconds⁻¹. It appears then, that an extremely large range of rate of shear is involved in the use of lubricants. In dispensing and in feeding from cups and boxes the rate of shear involved is usually in the comparatively low range from 0-1000 seconds⁻¹, and in the lubricating film from 1000 seconds⁻¹ to indefinitely large values."

Although many investigators have pointed out the dependency of the apparent viscosity of greases on the rate of shear, no test procedure measuring these characteristics is in general use. Consequently, greases are usually compared on the basis of the consistency determined by a penetration measurement, the accepted standard being the ASTM penetrometer using a double taper cone (2). This is in reality a hardness test at relative low shear rates, involving a very complex function of shear rates. The penetration value consequently cannot be translated to fundamental viscosity units for prediction of lubrication performance. For example, it

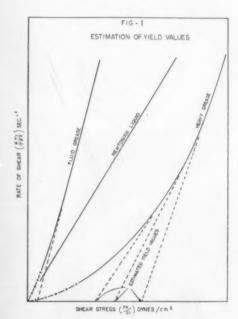
is possible to compound a given quantity of calcium soap into a 100 SUS/100°F oil, and also into a 1000 SUS/100°F, and obtain greases of identical penetration values. An attempt, however, to lubricate a heavily loaded, mine car wheel bearing with a representative high speed, textile mill grease containing the 100 SUS/100°F mineral oil is almost sure to lead to difficulty, even though the penetration values are the same. At best the penetration value serves as an index of the relative hardness or the amount of soap in a given type of grease, and for prediction of lubricant performance, the penetration results must be supplemented by knowledge of the viscosity of the mineral oil base stock used in the grease. This has often led to the use of the empirical rule that a lubricating grease for a given application should contain a mineral oil of the same viscosity as would be employed if the equipment were oil lubricated.

Other grease consistency tests, such as the Gardner and S. I. L. Mobilometers (3) used for semi-fluid lubricants, or the so-called liquid greases, also usually involve a variable and narrow range of rate of shear which complicates the application of the data to the solution of grease lubrication problems. Many so-called performance tests, such as low temperature starting and low speed running torque determinations on anti-friction bearings, gear trains, etc., may also be misleading because of failure to duplicate the rates of shear involved in actual service, or because of insufficiently wide range of shearing rates in the laboratory tests. For these reasons dispensing and consumption tests on greases usually are run on the actual equipment involved in the particular service. Further, the data cannot generally be applied to other dispensing or bearing equipment of the same type, but differing to a greater or lesser degree in design or size of dispensing orifice or bearing clearance, because of the difficulty or inability to reduce the data to fundamental units on the basis of which reliable comparisons can be made.

In an effort to obtain more reliable information on the consistency and the flow characteristics of greases, a number of investigators have employed viscosimeters capable of operating at different rates of shear (4, 5, 6, 7, 8). These include instruments utilizing parallel plates, rotating cylinders, and capillary tubes. The parallel plate

instruments, however, do not readily lend themselves to accurate measurements and the rotational or coaxial cylinder instruments, such as the Couette, MacMichael, and Stormer machines are applicable only to very thin or semi-fluid greases which will not channel. The capillary tube-pressure viscosimeters or plastometers consequently have been most frequently employed for investigation of the flow characteristics of greases.

The majority of the published results, however, have been obtained in gas actuated instruments, which permit only a limited range of rate of shear usually up to a few thousand reciprocal seconds rate of shear, whereas bearing lubrication frequently involves shear rates up to and including several hundred thousands. Flow rate determinations in the lower ranges of shear rate permit the estimation of the yield value or minimum force necessary to start flow (vield value). The data obtained from such measurements are undoubtedly of value in predicting the flow of soft greases from a lubricant reservoir to a bearing, and probably also to some extent the consumption and leakage of lubricant.



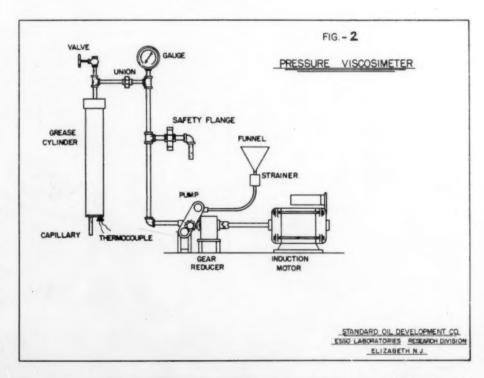
The interpretation of the test data obtained at low rates of shear, however, is complicated by the fact that the grease tends to slip along the walls of the capillary and extrude as a plug, thus indicating greatly reduced resistance to flow (9, 10, 11). As the shearing stress is increased, there is a gradual transition to the so-called telescopic, laminar, or viscous flow. The yield values extrapolated or estimated from shearing stress-shear rate curves, therefore, are not real and the mobility at low shear rates may vary

widely, depending upon the amount of slippage (illustrated in Figure 1). Several equations have been suggested for the purpose of correlating the data at low rates of shear, and their application to the experimental results has been reviewed in several publications.

Arveson in his study of the "Flow of Petroleum Lubricating Greases," (1) employed a constant-shear capillary tube viscosimeter which consisted essentially of a piston driven by means of a variable gear drive into a cylinder filled with the test material in order to force it through a capillary at a constant rate. This instrument offers a range of rate of shear sufficiently wide to cover the practical range of grease lubrication. Also, the design of the apparatus permits control of the rate of flow, and thus the shearing rate, with a given capillary. This is advantageous both from the standpoint of accuracy, and in that it permits the operator to predetermine the flow or shear rate at which a viscosity determination shall be made. This is in contrast to the usual procedure where the pressure is established and the rate of shear which varies with capillary dimension and the viscosity of the grease, is unknown. While the Arveson instrument has served admirably as a research tool for fundamental investigations of grease, it is too bulky and complex an instrument for general routine uses, and a simplified compact pressure viscosimeter is needed for grease viscosity determinations, not only for the solution of grease lubricating and dispensing problems, but also for control and specification purposes.

The Research Division of the Esso Laboratories some years ago investigated the flow characteristics of both oils and greases in a pressure viscosimeter similar to that developed by Arveson, and also in the low rate of shear, gas actuated type of viscosimeter, such as the Bingham plastometer (12). Data were collected on several series of greases of different types at relatively low rates of shear, and attempts were made to correlate the results with service performance. Apparent viscosity data obtained at elevated temperatures bore a relation to block grease consumption on slow speed bearings. Similar correlations were obtained on the dispensing of mine car greases, chassis lubricants and greases for spring actuated, Alemite, grease reservoir cups. However, field and simulated service tests in the laboratory, plus the knowledge available to the grease manufacturer of the type and percentages of soap employed, the viscosity of the mineral oil and manufacturing process controls appeared reasonably adequate.

More recently, however, the Imperial Oil Company, Ltd., Laboratories at Sarnia, in attempting to short cut laboratory consumption tests on automotive shackles succeeded in correlating consumption data with the rate of flow determined in a pressure viscosimeter of the Bingham type. Further studies by both the Imperial Oil Company, Ltd., and the Esso Laboratories of the Standard Oil Development Company have resulted in



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FIGURE 3

the design and development of a simplified constant flow rate pressure viscosimeter, which shows unusual promise for application to grease problems.

APPARATUS

The resulting instrument, which is diagrammatically illustrated in Figure 2 consists of a suitable capillary through which grease is forced from a cylinder by displacement with a hydraulic fluid delivered from a constant volume displacement, Zenith gear pump. The pump is driven by a one-third horsepower induction motor through a geared, constant speed reducer, and forces mineral oil, which is used as the hydraulic media, into the grease cylinder against the top surface of a piston which displaces the grease from the cylinder through the capillary into a receptacle. The photograph shown in Figure 3 gives a visual indication

of the size of the Zenith gear pump, and therefore serves to indicate the compactness of the entire pressure viscosimeter. Figure 4 gives the details of the grease cylinder and piston assembly. A Bourdon type pressure gauge, or for very low pressures, a mercury manometer is placed in the oil circuit to measure the pressure.

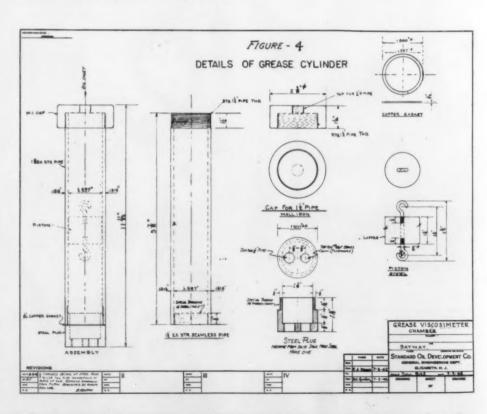
The Zenith pump which comprises the actuating mechanism in the S.O.D. pressure viscosimeter is designed to deliver a constant volume of fluid per revolution. Consequently, if the pump delivery rate is known, and its speed of rotation is accurately controlled, the volume rate of flow can readily be determined. The apparent viscosity can then be calculated if the pressure and capillary dimensions are known, by substituting these values in the Poiseuille equation: $\eta = \pi PR^4$,

8L v/t

and solving for η the viscosity coefficient in poises. P in dynes/sq. cm. is the pressure causing a flow of v/t cubic centimeters per second through a capillary of length L and radius R. The viscosity coefficient is defined as the ratio of shear stress to rate of shear ($\eta = F$), where the shear stress F is PR,

S 2I

and the rate of shear is 4 v/t. Thus, for a



given capillary the shear stress is a function of pressure alone, since pressure is the only variable; and the rate of shear is a function of the rate of flow, the other factors of the equation being constant.

The major sources of error in determinations with a pressure viscosimeter of this type are:

- 1. Rate of Flow Determinations
- 2. Determination of Capillary Dimensions
- 3. Measurement of Pressure
- Variations in Temperature of the Test Material

These sources of error and the means used to control them are discussed in connection with the detailed description of the viscosimeter and its operation.

RATE OF FLOW DETERMINATIONS

The induction type motor used as the power source maintains constant speed with variable loads providing the maximum capacity is not approached. The gear reducer, used to secure the desired pump speed, gives a 200 to 1 reduction, and thus also serves to minimize the effect of the load on the motor speed. The Zenith pump has a capacity of 0.584 cc. per revolution and with the present arrangement, employing a pump speed of 0.151 rev./sec., the rate of flow is 0.0882 cc./sec. The dimensions of the capillaries we have used for grease measurements are such that this rate of flow gives shear rates from 16 to 9100 sec.-1. The rate of shear may be further extended to some 15,000 sec.-1, by changing the 40-tooth gear between the gear reducer and pump to a 64-tooth gear. Other modifications such as using a variable speed transmission or a pump of greater volumetric capacity will extend the range greatly. The flow rate and range of shear rates developed with the present apparatus have been found suitable for a wide variety of lubricating greases without exceeding the delivery pressure limits recommended by the pump manufacturer. Calculation of the Reynolds numbers for the capillaries of relative small bore, that have been employed for measurements at the higher rates of shear indicate that their length can be reduced by one-half without inducing turbulent flow. This would greatly extend the rate of shear range obtainable without increasing delivery pressure.

The set-up was calibrated by determining both the volume and weight of the oil, and the weight of several greases delivered during measured time intervals while the pump was operating at fixed speeds. Some of the original calibration data were also obtained at several flow rates with oils of varying viscosity to investigate the possibility of pump slippage. It was found that a margin of error of 1% due to slippage could be obtained when using a 200 SSU at 210°F (bright stock) mineral oil for the complete range of allowable pressures, i.e., 0.1500 lbs./sq. in. This heavy oil, however, was found to be unsuitable for subzero temperature work due to the very large increase in viscosity, and to solidification in the grease cylinder. The method adopted to overcome this difficulty was as follows: The larger capillaries were used first in a series of measurements on a grease at low rates of shear with a 40 SUS/210°F low pour oil as the hydraulic media. At the low delivery pressures developed under these conditions the light oil is sufficiently viscous to prevent pump slippage. As the pressure increased on going to smaller capillaries, a more viscous oil was added to the pump intake to prevent slippage. The characteristics of the several oils used as hydraulic media and the range of delivery pressure over which they should be employed to minimize pump slippage are given in Table

(To be Continued)

TABLE II

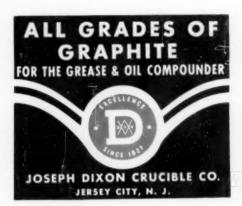
Pressure in	Capillary Radius (CM.)	Hydraulic Oil			
Cylinder Lbs./Sq. In.		Vis./210°F. (S.S.U.)	Pour, Approximate		
50-200	0.1908 0.123	40	-50		
200-500	0.0927	70	0		
500-1500	0.0760 0.0503 0.0231	200	+10		

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